

1919
J 52

THE UNIVERSITY
OF ILLINOIS
LIBRARY

1919
J 52

A STUDY OF COMPOUNDS OF SULPHUR WITH CARBON
STABLE AT HIGH TEMPERATURES

BY

EDWARD ARTHUR JEUDE

B. S. University of Illinois, 1918

THESIS

Submitted in Partial Fulfillment of the Requirements for the

Degree of

MASTER OF SCIENCE

IN CHEMISTRY

IN

THE GRADUATE SCHOOL

OF THE

UNIVERSITY OF ILLINOIS

1919

1919
552

UNIVERSITY OF ILLINOIS
THE GRADUATE SCHOOL

June 1919

I HEREBY RECOMMEND THAT THE THESIS PREPARED UNDER MY
SUPERVISION BY Edward Arthur Juede
ENTITLED A Study of Compounds of Sulfur with Carbon
Stable at High Temperatures.

BE ACCEPTED AS FULFILLING THIS PART OF THE REQUIREMENTS FOR
THE DEGREE OF Master of Science

E. A. Juede

In Charge of Thesis


W. A. Noyes

Head of Department

Recommendation concurred in*

Committee
on
Final Examination*

*Required for doctor's degree but not for master's



Digitized by the Internet Archive
in 2014

<http://archive.org/details/studyofcompounds00jeud>

TABLE OF CONTENTS

	Page
I. Introduction -----	1
II. Related Compounds-----	3
III. Method of Synthesis-----	4
(1) Heating sugar and sulphur in crucibles	
(2) Method of Analysis	
(3) Heating gilsonite and sulphur in crucibles	
(4) Passing sulphur vapors over gilsonite coke	
(5) Refluxing coke with sulphur	
(6) Pressure work	
(7) Removal of excess sulphur	
IV. Effect of Temperature, Time and Other Variables on the Formation and Stability of the Sulphur Compounds--	15
(1) Effect of Temperature on Normal "cokes"	
(2) Effect of Time on Normal "cokes"	
(3) Effect of Time and Temperature on "cokes" below saturation.	
(4) Character of Carbon.	
V. Variation of sulphur retained by coke from coal under variable conditions as to atmosphere-----	20
VI. Description of Apparatus-----	25
VII. Discussion and Summary-----	26
VIII. Appendix-----	28
IX. Bibliography-----	29

LIST OF PLATES

	Page
Plate I Apparatus used in making cokes by crucible method-----	5
Plate II Method of mixing charge-----	7
Plate III Apparatus used in making cokes by passing sulphur vapors over heated carbon----	11
Plate IV Apparatus used in refluxing carbon with sulphur-----	14
Plate V Curves showing effect of nitrogen on percent of sulphur retained by coke made from coal, first run-----	22
Plate VI Curves showing effect of nitrogen on percent of sulphur retained by coke made from coal, second run-----	24

ACKNOWLEDGMENT

This investigation was carried out in the Chemical Laboratory of the University of Illinois during the year 1918-1919.

It was undertaken at the suggestion of Professor S.W. Parr and carried out under his direction.

The writer takes this opportunity to express his appreciation to Professor Parr for the helpful direction and assistance he has given in this work.

I

INTRODUCTION

The present work was undertaken in order to demonstrate the existence and determine, if possible, the physical and chemical properties of one or more compounds of carbon and sulphur which would be stable at high temperatures.

Mr. A.R. Powell points out in his thesis that all the forms of sulphur in coal change in the process of carbonization to two general types;- "sulphide sulphur and another form in very stable combination probably with carbon".⁽¹⁾

This last form which constitutes the greater percentage of the total sulphur present in the coke was the subject of the present investigation.

The literature was singularly devoid of any reference to this particular problem except for the possible compound indicated in Powell's work.

In his work on the "Changes in the Forms of Sulphur in Coal on Coking" he showed that it was not an iron-sulphur or silicon-sulphur compound, but that it seemed to be a carbon-sulphur compound. He also showed definitely by coking of the phenol extract that a portion at least of the coke sulphur was organic both as to character and source. The treatment with nascent hydrogen^{also} indicated a probable organic form.

However, the activity of coke toward phenol and of its

nitric acid residue toward ammonia shows that the organic sulphur in coke is different from the organic sulphur in the original coal.

Since the method of attack in the previous work was chiefly analytical it was thought that if the synthetical method was employed more light might be thrown upon the problem.

II.

RELATED COMPOUNDS.

Before going into the present work it might be well to consider the various compounds of sulphur which appear as definite entities in the literature and have a possible bearing on this problem.

There are compounds of the general character $C_x S_y$, but all these are either gaseous, liquid or if solid, have melting points far below the temperature of coking. ⁽²⁾

There is also a compound of the general composition $C_x N_y S_z$, but its characteristics also eliminate it as a possible factor in the present investigation. ⁽³⁾

A search of the literature back to 1910 yielded nothing which had any bearing on the union of carbon and sulphur to give compounds stable at high temperature with the one exception of the compound indicated in Powell's work.

III.

METHOD OF SYNTHESIS

The first experiments of the present work were to find whether or not sulphur could form compounds with carbon other than those already known-, that is, whether a compound of carbon and sulphur could be synthesized which would be stable at high temperatures.

Since it had been shown that the compound under investigation was neither an iron-sulphur nor a silicon-sulphur compound, but probably a carbon-sulphur compound it was thought desirable to eliminate as many variables as possible and deal only with carbon and sulphur (i.e.) to work with the elements as nearly as possible and not with coal or compounds such as iron sulphide which would introduce other variables.

As a preliminary experiment five "cokes" were made by heating varying percentages of sugar and sulphur together in a 100 c.c. porcelain crucible, covered with a porcelain lid and heated over a #8 Meeker air blast as shown in Figure I. At first the crucible occupied the position indicated by the dotted figure and finally after the mass had coked sufficiently to prevent its forcing off the lid it was lowered to the second position. The heat at this point was so intense as to cause the ring which supported the nichrome triangle and crucible to sag about $1/4$ inch.

The crucibles were heated for 15 minutes in this second position after the mass had been broken into a relatively fine powder with a blunt glass rod.

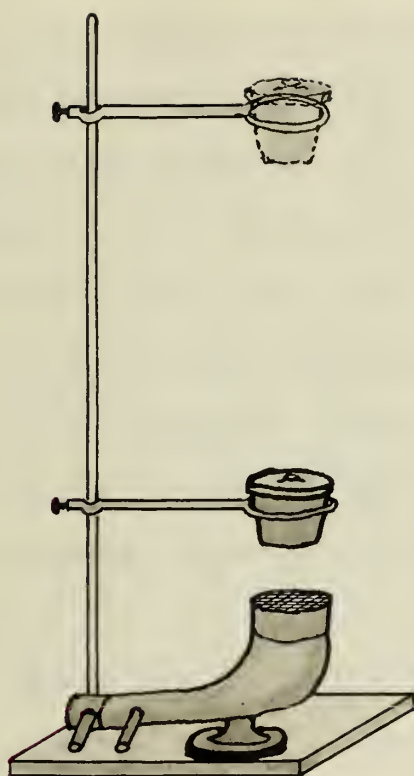


Plate I.

This procedure was followed in order to heat all of the material to the same temperature and drive off the excess sulphur.

The crucible was allowed to cool and the material was ground to pass a 40-mesh sieve and analyzed by the sodium peroxide method using the Parr sulphur bomb.

Quite a bit of trouble was encountered in fusing these coke substances so as to obtain complete fusions.

After a great many trials with various proportions of sample, sodium peroxide and potassium chlorate accelerator, it was found that better fusions were obtained with these cokes when the chlorate was omitted. However, poor fusion still resulted.

It was thought that since the coke consisted of only carbon and sulphur it might be well to add additional "fuel" which would raise the temperature of the reaction. With this in mind 0.2 gm. of sugar was introduced into the charge. This gave better results, but the cokes made at the higher temperatures still showed some free carbon after being fused.

The amount of sugar was gradually increased until finally the charge which gave best results was determined. This charge consisted of 0.5000 gm. sample, 1.00 gm. sugar and 1 measure (10 gm.) of peroxide.

This charge was adopted for all subsequent work and gave perfect fusions in every case, providing the charge was thoroughly mixed before closing the bomb.

The mixing was accomplished by holding a spatula in the right hand against the right side of bomb with the handle tilted slightly backwards and the face of the blade perpendicular to the side of



Plate II.

the bomb at the point of contact.

The bomb was rotated between the first two fingers and thumb of the left hand, allowing the bottom of the bomb to rotate on the side of the third finger as shown in Figure 2. By this procedure the material is elevated and drops into the space directly back of the spatula causing thorough mixing in about 25 rotations.

It was found that with thorough mixing of the charge as indicated perfect fusions resulted, but that poor mixing caused poor fusions.

The bomb was then assembled and after the cap had been tightened with the wrenches, the bomb was heated in a pointed blast lamp flame until the reaction started. The reaction was allowed to proceed for 6 - 8 seconds after which the bomb was plunged into cold water and allowed to cool partly. The bomb was then opened and the cup containing the fusion removed, placed in a beaker and covered with 150 - 200 c.c. water.

The mass dissolved readily if the bomb cup was still warm, but very slowly if the bomb has been cooled too much.

The bomb was removed and with the aid of a stirring rod, washed and enough concentrated HCl added to just give a clear yellow solution.

This was filtered to remove any solid material and the filtrate brought to boiling after dilution to 400 c.c. Ten cubic centimeters of hot 10% BaCl_2 was then added drop by drop with stirring and the solution was allowed to digest for at least one hour, after which it was filtered, washed three times with hot water containing

1 c.c. concentrated HCl per 100 c.c., then washed three times with pure hot water, dried, ignited and weighed.

The per cent of sulphur in these first five cokes was figured on the basis of the carbon in sugar and indicated simply that a relatively large per cent of the sulphur was retained even at the high temperature employed.

Gilsonite was substituted for the sugar and four cokes were made following the same procedure as before.

The material showed no further sign of coking after being ground and heated.

The results of this run were as follows:

Table No. 1

Cokes made by heating gilsonite and sulphur.

	% gilsonite	% sulphur	% S in "coke"
Coke #1	30	70	6.58
" #2	20	80	6.35
" #3	10	90	7.35
" #4	5	95	7.75

The results of this run were more satisfactory as they check fairly close considering the crude apparatus used.

The method of making these cokes was not satisfactory due to the swelling of the charge, the liberation of SO_2 , and other factors.

In order to prepare saturated compounds several new methods were employed.

The first of these was to pass sulphur vapors over heated gilsonite coke contained in a quartz combustion tube which was heated by four blast lamps. Heating with blast lamps was unsatisfactory and electrical heating was substituted. The apparatus used is shown in Figure III. The generation of sulphur vapors was adjusted so as to prevent them from pouring into the laboratory, the object being simply to supply an atmosphere of sulphur over the heated carbon.

The "cokes" made were used in later experiments and will be considered as they were used.

In order to prepare larger quantities more conveniently than was possible by any of the methods so far used, a new scheme was tried; namely, that of refluxing the carbon with sulphur. This was accomplished in the apparatus shown in Figure IV.

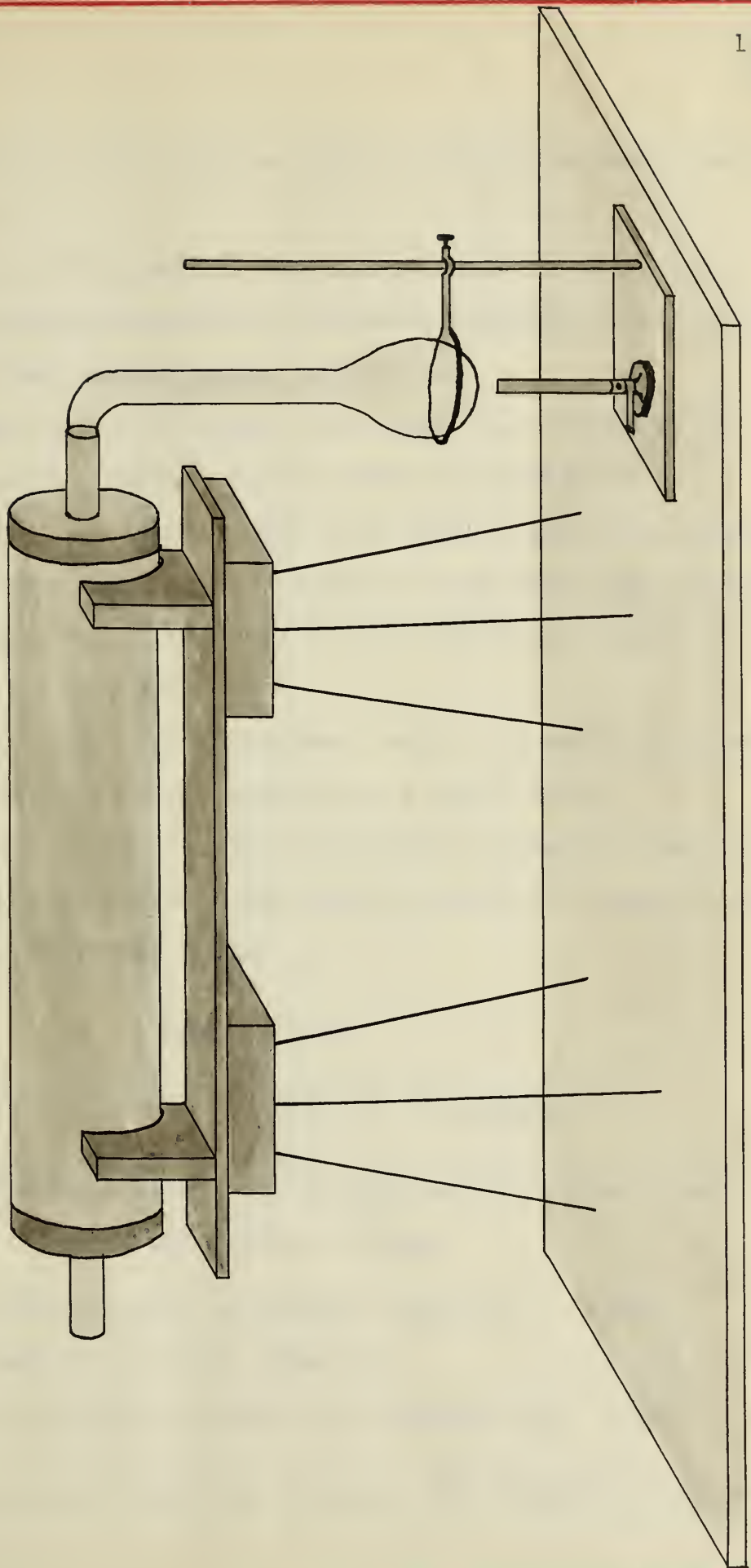
This was by far the best of any tried and was used for all subsequent work.

Pyrex and other glass ware was used for the lower temperatures, but fused silica ware was employed for the higher temperatures.

An attempt was made to form this material under pressure by heating carbon and sulphur together in Carius tubes and metal containers, but the bombs blew up in each case. In the metal bombs, the material was so badly attacked by the sulphur as to decrease the strength of the container and contaminate the material formed.

This method was consequently abandoned.

In every case the "coke" as formed had an excess of sulphur; that is, some free sulphur present which had to be removed before

Plate III.

analysis in order to find the per cent of sulphur actually held by the carbon.

The removal of this excess was accomplished in the earlier experiments by simply heating in a covered crucible on the assumption that any free sulphur would volatilize.

It was found that this excess could also be removed by extraction with carbon bisulphide or carbon tetrachloride.

The extraction was carried out in a Soxhlet extractor using a 2 foot water-cooled condenser. A jet of live steam was directed against the extractor at the base of the thimble and adjusted so as to keep the solvent hot.

It was also found that the excess could be removed by passing an inert gas over the heated sample for a short time.

In every case it was possible to obtain a substance which on analysis showed approximately the same per cent of residual sulphur as shown in the following table.

Table No. 2.

Combined sulphur after removal of excess.

Coke made by passing sulphur vapors over heated carbon in gas heated combustion furnace.

(1) Coke heated for 15 min. in covered crucible	-	6.8%
(2) " extracted " 1 1/2 hrs. with CS_2	-	6.26%
(3) " heated at 500°C in stream of nitrogen 1 Hr.		6.25%

The inert gas used was Tank nitrogen (Air-Reduction Co.) which

was purified by passing it thru absorption bottles, two of which were filled with alkaline pyrogallol solution and the last with concentrated sulphuric acid.

On analysis the gas issuing from the absorption train showed 100% nitrogen on 100 c.c. sample.

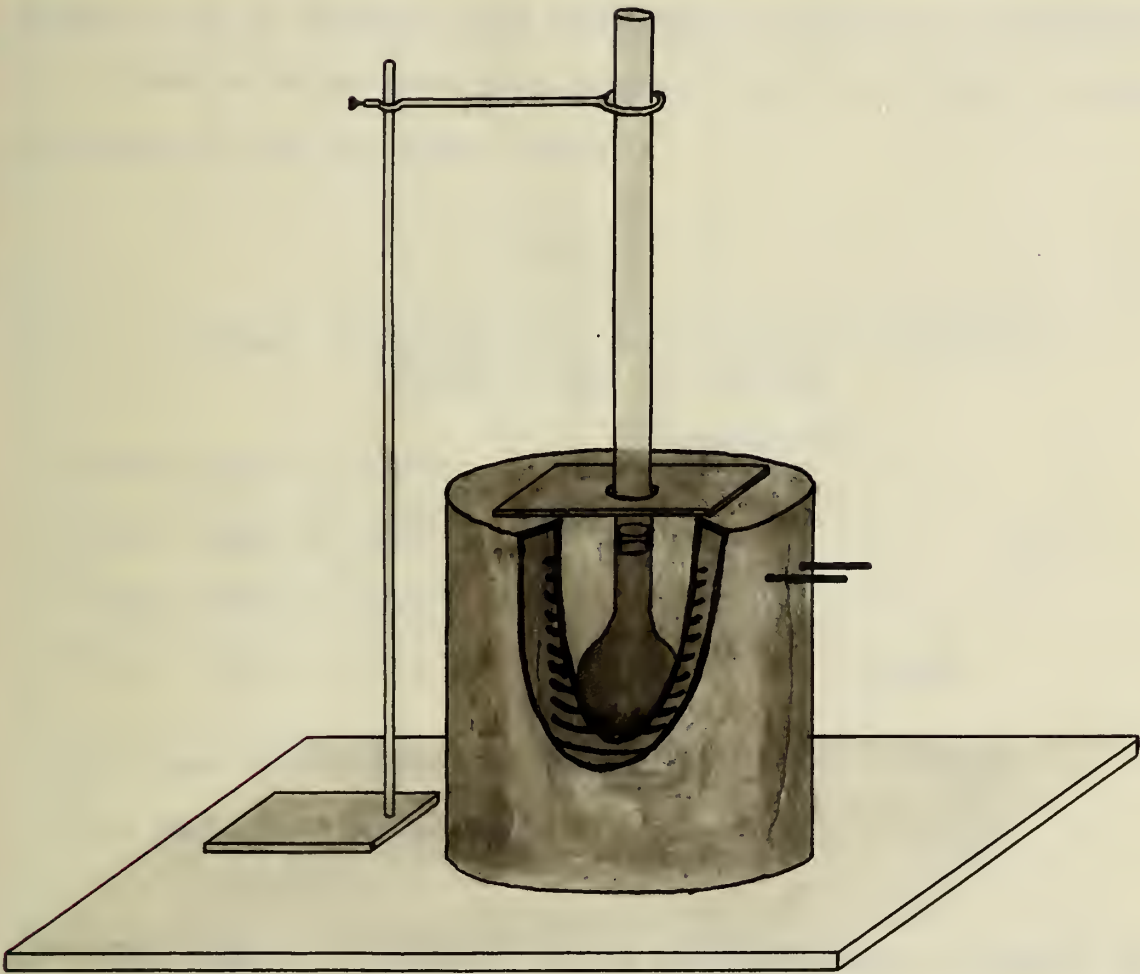


Plate IV.

EFFECT OF TEMPERATURE, TIME AND OTHER VARIABLES ON THE FORMATION AND STABILITY OF THE SULPHUR COMPOUNDS.

In the effort to remove the excess sulphur and in the synthesis of the "cokes" themselves, certain variables were inadvertently introduced which were conducive of interesting results. The first of these variables was temperature. The variation in the per cent of residual sulphur in the different cokes raised the question as to whether this temperature factor was responsible.

Certain cokes were then compared and the effect of temperature is shown in the following table.

Table No. 3.

Effect of Temperature on Per Cent of Sulphur.
Taken up by the Carbon

	Residual S.
"Coke" made at 750°C - 800°C.	6.25%
"Coke" made at 850°C - 900°C.	5.80%
"Coke" made at 1000°C.	2.40%
"Coke" made at 1100°C.	1.77% - 1.88%
Coke made from high sulphur coal at 1160	- 3.18%
Coke made from high sulphur coal at 1260	- 2.92%

The effect of temperature on decomposition of these cokes is not noticeable so long as they are in a closed crucible or in an atmosphere of sulphur as they are stable under these conditions, but it is very noticeable when an inert gas is passing over or thru the material. This is shown in the following table which is made up of data from experiments in which this last condition was met.

Effect of Temperature on Decomposition of Cokes
Residual S.

Coke made at 750-800° C, heated 1 Hr. at 500° C	- 6.25%
" " " 750-800° C, " 1 Hr. at 700° C	- 4.80%
" " " 1100° C " 1 Hr. at 500° C	- 1.71%
" " " 1100° C " 1 Hr. at 900° C	- 1.58%
Coke made from high sulphur coal at 1160° C	- 3.18%
" " " " " " at 1260° C	- 2.92%

The temperature at which carbon would combine with sulphur was also investigated to some extent and was found to vary from 300° C to 1100° C which were the limits of temperature tried.

This is shown by a study of the following table along with those previously shown.

The cokes in the following table were made by heating sugar carbon and gilsonite carbon respectively with sulphur in glass flasks in an electric furnace for different lengths of time then extracted with CS₂ or CCl₄ for 24 hours in Soxhlet extractor as previously described.

Table No. 4a

Effect of relatively low temperature (300°C)
on combination of Carbon and Sulphur

				Residual Sulphur after extraction
"Coke" made by heating sugar C and S for 50 Hrs.				
" " " " " at 300° C extracted CS ₂				---- 14.29% S
" " " " " C and S for 50 Hrs.				
" " " " " at 300° C extracted CCl ₄				---- 10.01% S
" " " " " C and S for 12 Hrs.				
" " " " " at 300° C extracted CCl ₄				---- 5.03% S
" " " " " gilsonite C and S for 12 Hrs.				
" " " " " at 300° C extracted CS ₂				---- 11.72% S

This experiment was run simply to see if carbon could combine with sulphur at this temperature and indicates that it does. It also raises the question as to whether CS₂ extracts all the excess sulphur as previously assumed. The relative value of CS₂ and CCl₄ as a solvent should be further investigated.

Effect of Time

In studying the properties of the hypothetical sulphur-carbon compound it seemed desirable to determine the effect of time on the amount of sulphur that might be taken up by the carbon.

Various cokes were tested in the course of the investigation by heating in the furnace shown in Fig.III and passing nitrogen gas over them as explained under the removal of excess sulphur.

The effect of time on the loss of sulphur under these conditions is given in the following table.

Table No.5

Effect of Time on the loss of Sulphur.

"Coke" used above made by refluxing gilsonite with sulphur

for two hours at 1000° C

"Coke" heated for 1 Hr. at 450° C -----1.81% S.

"Coke" heated for 7 Hr. at 450° C -----1.87% S

"Coke" " " 1 Hr. at 500° C -----1.77% S

"Coke" " " 7 Hr. at 500° C -----1.68% S

The results of this experiment seem to indicate that the time element has no effect after a relatively short time (i.e.) that the loss of sulphur is not materially increased by six hours additional heating under the conditions indicated. This experiment with another in which samples of a "coke", made by refluxing gilsonite coke with sulphur at 1100° C, were heated in the furnace with nitrogen gas passing over them keeping conditions the same except temperature,

seemed to show that time and temperature had practically no effect on samples which were below a certain saturation. That is if we assume that at any given temperature, for instance 800° C, carbon can take two to three percent of sulphur then heating at a lower temperature (e.g.) 500° C for any time up to seven hours seems to have no effect on a "coke" which contains less than this percent of sulphur.

This is shown in the following table:

Table No.6

Effect of Time and Temperature on "Cokes" below saturation							Residual Sulphur
"Coke"	made at	1100° C	heated for	1 Hr.	at	500° C	--1.67% S
"	"	"	"	"	"	1 Hr.	" 600° C--1.62% S
"	"	"	"	"	"	1 Hr.	" 700° C--1.58% S
"	"	"	"	"	"	1 Hr.	" 750° C--1.50% S
"	"	"	"	"	"	1 Hr.	" 900° C--1.59% S
"Coke"	"	"	1000°	"	"	1 Hr.	" 450° C--1.81% S
"	"	"	"	"	"	7 Hr.	" 450° C--1.87% S
"	"	"	"	"	"	1 Hr.	" 500° C--1.77% S
"	"	"	"	"	"	7 Hr.	" 500° C--1.68% S

Character of Carbon

Amorphous or coke carbon is quite a variable in its own right and while no definite attempt was made to determine the nature of this factor, it was found that while sugar and sugar carbon, gilsonite and gilsonite carbon would all combine with sulphur under the conditions of the experiments tried, graphite would not combine with the sulphur even when exposed to sulphur vapors at temperatures up to 900° C for several hours. Since amorphous carbon is so variable depending not only on the source from which it is derived, but also on how it is prepared, it will be necessary to determine just how this variableness affects the amount of sulphur taken up before comparable results can be obtained and a clear insight into the present problem arrived at. This phase of the subject must be left for further investigation.

V

Variations of Sulphur retained by Coke from Coal
Under Variable Conditions as to Atmosphere

From the results of the foregoing work it seemed that a stream of inert gas such as nitrogen was able to reduce the percent of residual sulphur held by the carbon and new experiments were undertaken to find the effect of the nitrogen on the percent of sulphur retained by the coke made from coal.

With this end in view 75 gms. of a high sulphur coal was crushed to $1/8$ " size, and introduced into a 5" assay crucible fitted with a perforated false bottom thru which a $1/8$ " diameter quartz tube passed, and a cupelling dish as a lid.

This crucible was then set into an electric pot furnace and heated at the desired temperature for two and one half hours.

Two sets of cokes were made, the first without passing nitrogen thru the quartz tube, the second with nitrogen passing thru the tube up thru the false bottom and thru the mass.

The temperature was taken with a Leeds and Northrup Pt., Pt.-Rh couple enclosed in a quartz tube which was placed as near the center of the charge in the crucible as possible.

All conditions were held as nearly constant as possible except the temperature which was varied from 600° to 1100° C for the cokes made without nitrogen and from 710° C to 1000° C for the cokes made with nitrogen.

The results of this experiment are given in the following table and shown graphically on Plate V.

Table No. 7

Effect of Nitrogen on percent of sulphur retained by coke
made from coal.

Without Nitrogen		With Nitrogen	
<u>Temp.</u>	% S in Coke	<u>Temp.</u>	% S in Coke
600° C	6.14%	710° C	4.54%
810 - 820° C	4.94%	800° C	4.58%
900° C	5.85%	1000° C	4.23%
1100° C	4.92%		

The results of this experiment seem to show that the percent of sulphur in the coke is reduced by passing nitrogen thru the mass while it is being coked.

In order to study this further the method was refined in order to get better temperature control.

This was done by heating small samples (2 1/2 gms.) in porcelain combustion boats in the combustion furnace perviously used.

In this experiment twelve samples were made, six without the use of nitrogen, and six while passing nitrogen thru the furnace during the coking.

The results of this experiment are given in the following table and shown graphically on Plate VI.

Temp. °C.

1100

1000

900

800

700

600

Plate V.

Without Nitrogen

With Nitrogen

Percent Sulphur.

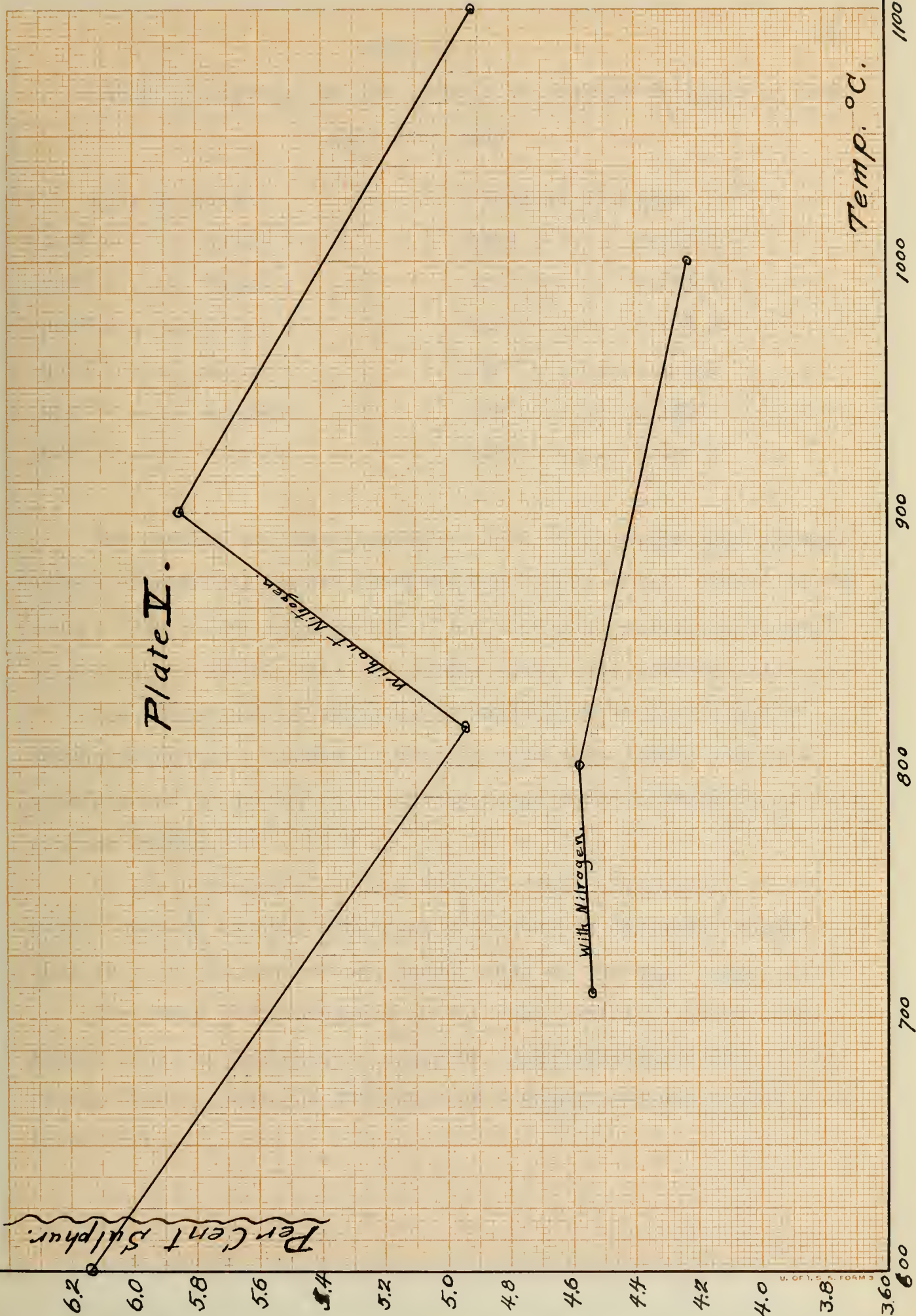


Table No.8

Effect of nitrogen on the percent of sulphur retained by coke
made from coal

With Nitrogen	Without Nitrogen
640° C ----- 4.32% S	650° C ----- 4.98% S
700° C ----- 3.60% S	700° C ----- 4.54% S
800° C ----- 4.56% S	800° C ----- 4.16% S
850° C ----- 4.41% S	850° C ----- 4.21% S
950° C ----- 4.05%SS	950° C ----- 4.15% S
1000° C ----- 4.22% S	1000° C ----- 5.42% S

The results of this experiment seem to show the same general effect of nitrogen as the previous experiment showed except in the case of the cokes made at 800° C and 850° C in which the percent of sulphur is higher than in similar cokes made without nitrogen.

The conditions of these experiments must be more rigidly standardized and a number of similar runs made before any conclusions can be drawn, as to the apparent erratic behavior of this coal on coking.

It is interesting to note however, that the percent of residual sulphur in coke made from this coal is decidedly higher than that in the synthetical "coke" made at the same temperature.

One other fact developed in the examination of these cokes, namely, that an appreciable quantity (approximately 1-2%) of the finely divided coke was attracted by a magnet and was probably reduced iron.

Temp. °C.

900

800

700

600

Plate VI.

Per Cent Residual Sulphur.

5.5

5.2

5.0

4.8

4.6

4.4

4.2

4.0

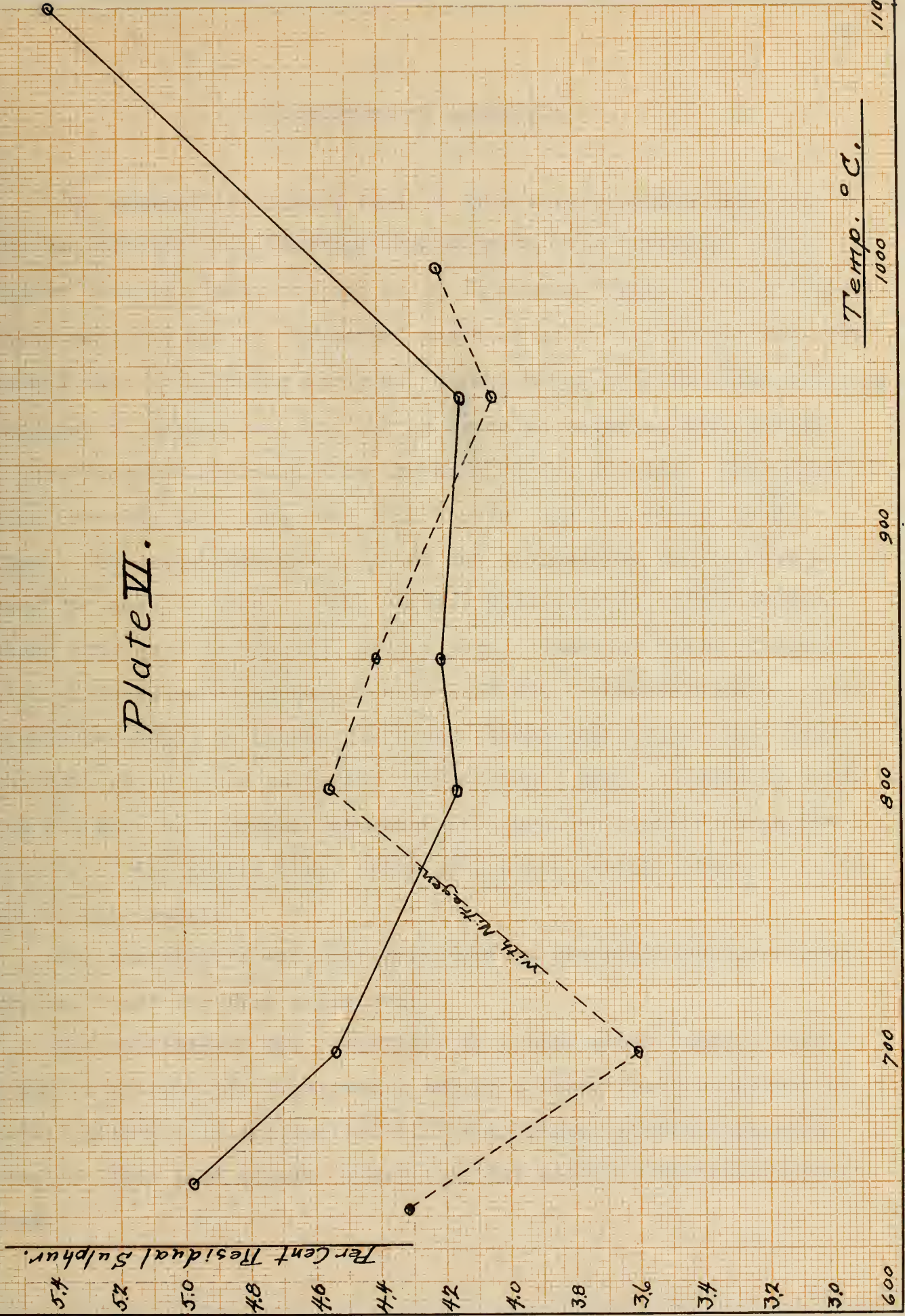
3.8

3.6

3.4

3.2

3.0



VI

DESCRIPTION OF APPARATUS

The combustion furnace used in this investigation, Fig.III, was constructed from standard 85% magnesia pipe covering, a 3/4" quartz tube two feet long and No.14 IA"Chromel" wire. Two circular disks were cut out of "Transite" board so as to fit snugly into the recess made by allowing the brass straps which held the pipe covering together to extend over the end of the pipe covering thus giving a more finished appearance to the ends of the furnace. Two sizes of pipe covering were used, one for 1" pipe ,and the other for 3" pipe. The large size was hollowed out slightly so as to accommodate the smaller size. This furnace when mounted on the wooden-stand indicated in Fig.III, gave a very convenient piece of apparatus as it allowed of quick renewal of the heatingelement it being simply necessary to unhook the brass strips and replace the burned-out coil which could be easily replaced by a new coil made by winding #14 wire on a mandrel of about 1/8" smaller diameter than the outside of the quartz tube. This gave a coil which slipped over the quartz tube easily.

The temperature was regulated by wire Rheostats one for coarse, and the other for fine adjustment.

The temperature was determined by a base metal Hoskins Pyrometer in the earlier experiments and by a Leeds Northrup potentiometer indicator in conjunction with a platinum, platinum-rhodium couple. This last apparatus was accurate within five degrees centigrade.

VII

DISCUSSION AND SUMMARY

The present work gave no really definite indication as to whether sulphur was held by the carbon in chemical combination or simply as absorbed or adsorbed material, but many interesting facts developed and several lines of attack seemed to be indicated. These are given in the appendix.

The sulphur seems to be held in such a way as to resist the action of ordinary extractions, but it would be interesting to try the effect of phenol and other solvents under pressure. Fusion with sodium or potassium hydroxide or other molten solvents would probably be well worth while if a method of separating the solid residue either directly on a porous plate or by filtration after dilution can be worked out.

If by any method a substance could be isolated from the coke which could then be studied along with the hypothetical carbon-sulphur compound it would probably give a clearer notion as to the way in which sulphur is held by the carbon.

The following summary of conclusions seems to be warranted from the data as given:-

SUMMARY

- (1) Carbon seems to have an avidity for sulphur at high temperatures.
- (2) Heated carbon which has been subjected to sulphur fumes retains an appreciable quantity of sulphur upon cooling.
- (3) A certain percentage of the sulphur is combined with the carbon in such a way as to resist the action of solvents or heating in a current of inert gas as distinct from the excess sulphur which is removed by this treatment.
- (4) The amount of sulphur taken up by the carbon seems to depend on the refluxing temperature, less sulphur being absorbed at the higher temperatures.
- (5) The decomposition of the "cokes" or the loss of sulphur by the carbon when heated in a stream of nitrogen seems to increase with increase of temperature.
- (6) "Cokes" made at high temperatures are relatively stable at low temperature when heated in a stream of nitrogen.

VIII
APPENDIX

During the course of the investigation, a number of interesting experiments suggested themselves, and it was thought that if these were given in outline form they might prove helpful for future reference.

(1.) Try solvent action of fused Na_2CO_3 on "Cokes" in atmosphere of CO_2 and analyze water solution for any possible sulphur compounds such as H_2S , SO_3 , SO_4 , HCNS etc. before oxidation takes place.

(2) Analyze core and crust of cokes from coal, when coked under conditions of experiments under V, for ash and sulphur to find distribution of sulphur and ash.

(3) Try to synthesis the hypothetical Carbon-sulphur compound using sulphur compounds such as H_2S , SO_2 , FeS , etc. in place of sulphur.

(4) Find how the sulphur is given off when heated in a stream of nitrogen. (i.e.) as sulphur or as a compound.

(5) Prepare "cokes" at different temperatures until saturation is complete and find relation of temperature to percent of residual sulphur in "coke" after removal of excess. Then heat a compound made at low temperature (500°C) at higher temperatures 600°C , 700°C etc. and find relation of temperature to percent of residual sulphur in "coke" after removal of excess. Plot results.

(6) Find if percent of iron or nitrogen in sample affects percent of sulphur taken up by carbon containing various percents of these elements.

BIBLIOGRAPHY.

(1) Univ. of Ill. Eng. Exp. Station, Bull. No. 111, 1919.

(2) Löw, O., Amer. J. Science (2), 41, 251

Sidot, Comp. Rendu. 74, 180.

81, 32.

(3) Muthmann, W., and Clever, A., Zeit. Anorg. Chem. 13, 203, 1897.

UNIVERSITY OF ILLINOIS-URBANA



3 0112 079828486